DIMENSIONS NBS

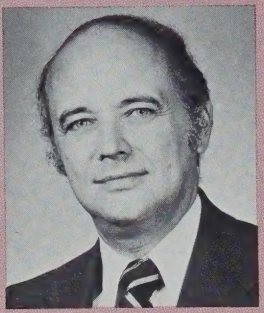
The magazine of the
National Bureau
of Standards
U.S. Department
of Commerce

May/June 1980

THE SOUNDS OF FAILURE. See page 12.

COMMENT

POLYMER SCIENCE AND STANDARDS



In the seven decades since Leo H. Baekeland introduced the first commercial synthetic plastic produced entirely by the reaction of small molecules, polymers have left little of our culture and technology untouched. We have moved rapidly into an age in which an overwhelming number of humanity's needs are served by polymers. The increase in production and use has been spectacular; the volume of polymers produced last year exceeded that of steel. Recent summaries show that polymers already account for over one-third of the total industrial research and development expenditure for metals, polymers, and inorganic materials. Polymers also constitute a similar proportion of the value added by manufacture (a measure of the relative economic importance of manufacturing among industries) and the number of scientific publications.

Polymers have been shaped by intellectually sophisticated research which has been recognized by a number of Nobel prizes. The resulting materials have contributed to national productivity through their economy, ease of fabrication, and useful properties such as low density, corrosion resistance, and toughness. They are energy efficient, using an average of one-fourth the energy per unit volume required by metals for the conversion from raw to refined material and yielding further savings in processing and shipping.

Polymers have entered increasingly sophisticated and demanding applications. Poly (vinylidene fluoride) replaces electromagnetic transducers in high fidelity systems. Structural parts of aircraft are composites with a polymer matrix. Polyurethane elastomers are used to pump blood in artificial heart assist devices. The list of such examples is very long, encompassing trends for the increased use of polymers in packaging, construction, transportation, energy, electronics, information handling, clothing, appliances, dentistry, and medicine. Since polymer technology is on the steep upward part of the learning curve, these trends create new needs for concepts, measurements, standards, and data that can be used to insure reliable performance in use, processing, and other phases of the material cycle.

To identify the key needs, the managers of the Polymer Science and Standards Division make structured visits to industrial and other Government laboratories. Recent visits have included Bell, Du Pont, Army R and D Command, General Motors, Boeing, and Lawrence Livermore. Surveys concerned with areas such as durability, processing, and composites are conducted. These and other contacts with experts in industry, trade and professional associations, government, and academia insure that the Division's efforts are directed toward fostering the effective use of polymers in solving national problems cited by the Director as appropriate for NBS. These include the growth of industrial productivity, prudent utilization of energy sources, a reasonable system for regulation of the effects of technology, and a wise policy of materials utilization.

The Division's activities are grouped into areas concerned with mechanical reliability in load-bearing applications, chemical durability and lifetime prediction (including the migration of protective additives), characterization methods and standards for molecular composition and use properties, dielectric properties and electrical conduction, and reliable dental and medical materials. Substantial cooperative efforts are conducted with the Food and Drug Administration, the Department of Defense, the National Institutes of Health, the American Dental Association, the Department of Energy, the Bureau of Engraving and Printing, and others. The cooperative effort with the Bureau of Foods to produce methods for predicting the rates of migration of small molecules from plastic containers into foodstuffs is described in the DIMENSIONS feature on Additive Migration in Plastics in this issue.

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INTERFACE

UPDATE

WRAPPING IT RIGHT

EVALUATING ADDITIVE MIGRATION IN PLASTICS

by Gail Porter

When broccoli cooks in a plastic pouch,
And butter spreads from a plastic tub;
When bologna pops from a plastic pack,
And cheese is dressed in plastic wrap;
When plastic bags carry lunch to school,
And plastic bottles keep the cola cool;
When plastic protects the chicken wings,
And plastic cups hold Singapore slings;
When all of this has come to be,
It's time to step back thoughtfully
And ask, "Are we still eating just the food?
Or are we eating the plastic too?"

TODAY you, like just about everyone else in the United States, probably ate something that had been packaged in plastic. In the past 10 years, the use of plastic in food packaging has risen dramatically, and some package experts predict that plastic will capture the majority of the packaging market within the next few decades. (According to the Society of the Plastics Industry, the use of plastic packaging materials is a major reason that the United States has the lowest food spoilage rate in the world.)

It is easy to see why plastics are so popular. Their versatility seems almost limitless—there are firm plastics, stretch plastics, plastics that bounce, plastics that stick, molded plastics, and just plain plastics. Moreover, plastics can be made more durable than paperboard and are lighter in weight than metals.

But plastics, like other materials, are seldom perfect for everything. Components of plastics, organic materials, can mix with other organic things—like food.

Plastics consist of single molecules or monomers, combined into extra long chains to produce a polymer. However, some single molecules or smaller groups of molecules called oligomers, for one reason or another, fail to hook up in the chain. All plastics also contain "additives" which are sprinkled into a brewing batch of plastics, like herbs into a stew, to obtain the desired properties such as heat resistance, rigidity, color, or durability.

Because these smaller molecular groups and additive compounds are not firmly hooked up in the polymer chains, they can migrate from the plastic. Naturally, if the additives are removed from the plastic, the property they imparted will be removed as well. The package may fade, become brittle, or even lose its shape. And, if these compounds are moving out of the polymer, they must be moving *into* something else. When that something else is food, the possibility of contamination arises.

It is the business of the Food and Drug Administration (FDA) to assure that the food we buy is safe to eat. This includes requiring plastics manufacturers to prove—through a battery of scientific tests—that materials used in packaging do not contaminate food with dangerous amounts of toxic substances.

These tests may include analytical studies to determine how much, if any, of the monomers, oligomers, or additives in the package will migrate, as well as toxicological studies to determine if the projected release of these substances could be harmful to humans or animals.

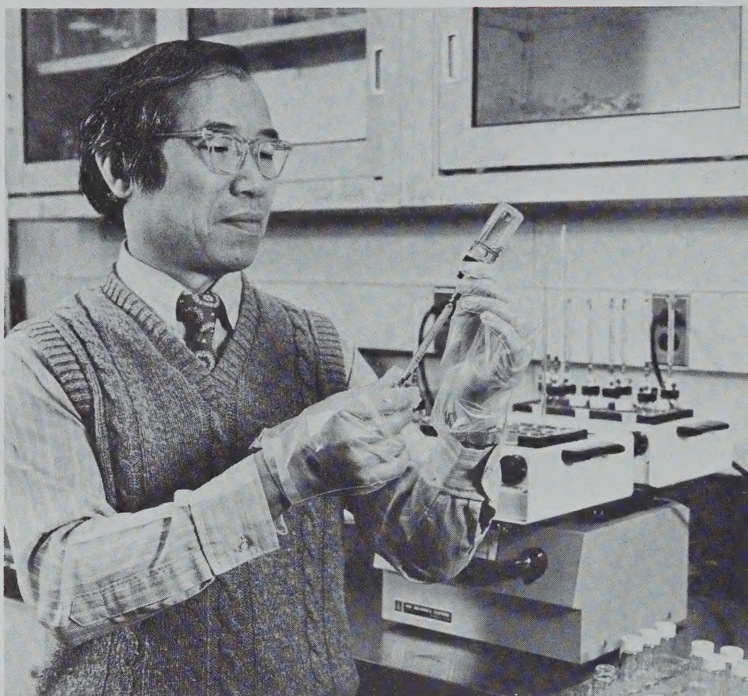
Tests like these are not cheap. According to recent estimates made by a representative of one major plastics company at a Packaging Association meeting, the process of developing and testing a totally new plastic packaging material could take anywhere from 5 to 10 years at a cost of $200,000 to $2 million, depending on the amount of migration occurring and the circumstances of the particular case.

Terry Troxell, a consumer safety officer of the Food and Color Additives Division of the FDA, points out that when only small variations from previously cleared combinations are involved, the time for regulation may be as short as one year.

In either case, both the FDA and the plastics industry would benefit if migration testing could be accomplished with less expenditure of time and money. So at FDA's request, scientists in the Polymer Science and Standards Division of the National Bureau of Standards are trying to trim the effort spent on polymer migration tests by learning more about the migration process itself. If the interactions between various sizes of molecules and types of polymer were better understood, they reason, it might be possible to eliminate some of the case-by-case testing now necessary without compromising public safety.

The regulation process as it stands now, says Leslie Smith, leader of the Polymer Stability and Standards group, is "tremendously inefficient. The Food and Drug Administration has asked us to develop the scientific basis of a more generalized approach."

Porter is a writer and public information specialist in the NBS Public Information Division.



Small samples of polyethylene with radiolabeled compounds are immersed in food-simulating solvents and monitored for migration. Project leader Shu Sing Chang uses a hypodermic needle to draw a sample of solvent for testing.

The NBS research group is searching for physical models that can be used to predict migration behavior. What physical factors are most important in governing migration and how can the relationship between these factors be expressed quantitatively?

In searching for the answers, the group has balanced its work between experimental measurements of additive migration and theoretical modeling. The result has been the development of equations and concepts which help put the complex nature of migration into clearer perspective.

In developing these rules, the researchers first needed to establish the crucial variables involved. For the most part, says Smith, the important considerations are:

1. The physical state of the polymer. Is it a glassy, semicrystalline or rubbery, amorphous type of polymer? Additives and oligomers can move as much as a million times faster through a rubbery, amorphous polymer than through a rigid, glassy polymer.

2. The type of additive. Larger molecular weight additives and oligomers generally move more slowly than smaller ones. Is the additive chemically like or chemically unlike the polymer? The less the compound resembles the polymer the more quickly it will move through it.

3. The type of solvent used to simulate the food. What kind of food will the package hold? Fatty foods like oils will cause faster migration in some types of plastics, whereas water will enhance migration in others. Liquids will dissolve more additives and oligomers than solids will.

4. The temperature, shape, and size of the package. Higher temperatures increase molecular movement and promote migration. The thickness of the plastic package and the amount of surface area in contact with the food will limit how much migration is physically possible.

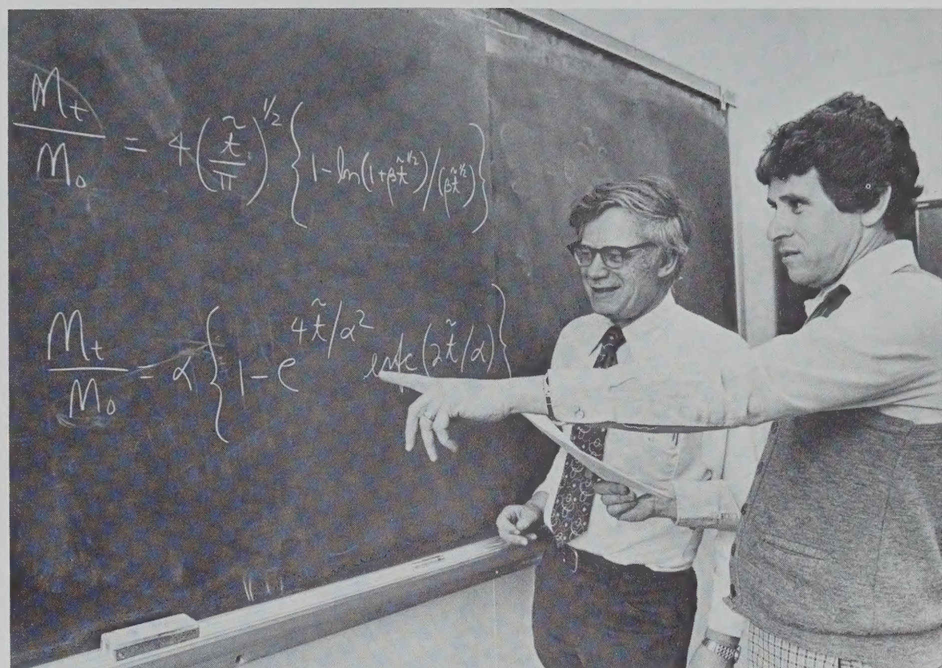
In setting up experiments to measure migration, NBS researchers first tested the semicrystalline polymer, polyethylene, because this is the type used in 65 percent of the plastic packages made in the United States.

Samples of a standard, well-defined polyethylene were infused with one of three different radiolabeled compounds—an additive called BHT and two different sizes of oligomers, n-octadecane ($C_{18}H_{38}$) and n-dotriacontane ($C_{32}H_{66}$). The compound BHT is frequently added to food and packaging materials to scavenge oxygen before it has a chance to oxidize the food or plastic molecules, causing decay. The two oligomers are common byproducts of the polyethylene manufacturing process.

By using radiolabeled compounds, where radioactive carbon 14 has been substituted for a certain percentage of stable carbon atoms, the migration behavior of the compounds can be tracked precisely, says Shu Sing Chang, a research chemist who is the project leader for these experiments.

Because real foods spoil and the behavior of pure chemicals is more predictable, the NBS migration tests rely on a number of different food-simulating chemicals as solvents. Mixtures of water and ethanol simulate alcoholic beverages, while heptane and trioctanoin mimic fatty liquids. Corn oil is used to check the differences in migration behavior between the pure chemicals and the real thing.

Small samples of the polyethylene are placed in the solvents and agitated in a reproducible way. At regular intervals, says Chang, samples of the solution are removed and tested for the radiolabeled compounds.



Right. *Theoreticians Isaac Sanchez (right) and Frank McCrackin discuss equations used in predicting the time dependence of additive migration.*

Using an instrument called a liquid scintillation counter, the researchers can detect as little as one-tenth of a nanogram (1×10^{-10} gram) of the additive or oligomers. The carbon-14 atoms in the compounds decay, giving off beta particles. The energy of the beta particles is absorbed by special organic molecules in the liquid and converted to light. The emitted light is then detected with sensitive photomultipliers.

These measurements are taken at repeated intervals until a state of equilibrium is reached and the level of additive or oligomer in the solution remains constant. It may take, says Chang, anywhere from a few days to more than a year for the solution to reach equilibrium.

While Chang and his group provide baseline data with his extremely accurate technique, NBS research chemist George Senich is working on an alternative migration measurement method for estimating the diffusion rate of some small molecules through polymers. Senich is working with a technique called inverse gas chromatography Unlike conventional gas chromatography where a compound to be analyzed is passed through a particular type of packed column, in this case the compound of interest is used to make the column, and a probe gas is passed through it.

Tiny sand-sized spheres of glass are first coated with polyethylene and then packed into a standard chromatography column. A flow of helium gas is pumped through the column and carried along with vaporized molecules of a solvent or oligomer. As the molecules move through the column, they make contact with the coated beads and diffuse into and out of the plastic before being swept into a detector.

With very slow gas flow rates, a point of equilibrium is reached when just as many molecules are diffusing into the beads as are diffusing out of the beads. The time needed to establish equilibrium tells something about the thermodynamic interactions between the two substances and can be used to estimate the quantities needed to predict migration behavior in real life situations.

At high gas flow rates equilibrium is not achieved, but an estimate of the diffusion rate of the solvent or oligomer in the plastic can be determined.

Left. As part of his fluorimetry experiments, research chemist Francis Wang checks the temperature of a polyethylene sample.

Walter Pummer (right) sets the liquid scintillation counter to measure the amount of radiolabeled additives and oligomers in a number of different solvent samples; John Maurey uses a computer terminal to analyze the results of previously collected data.

A final way NBS researchers are measuring migration is through the use of fluorimetry. Research chemist Francis Wang first immerses thin sheets of polyethylene in a well stirred solvent. Periodically the resulting solution is inserted into a closed chamber through which ultraviolet light of a very specific frequency is passed. The ultraviolet light excites relatively large organic compounds like antioxidants so that these molecules emit fluorescent light in a characteristic range of frequencies. The amount of light detected tells how much antioxidant has migrated out of the plastic.

Another use of fluorimetry, which Wang plans to initiate soon, involves taking periodic "snapshots" of a cross section of the polymer sheet. Using a microfluorimeter to detect the same characteristic fluorescence, he hopes to be able to detect the amount of antioxidant molecules at various locations within the polymer. By making several measurements of the same polymer at different times, he should be able to show how the concentration profile of the antioxidant changes over time as the molecules migrate (toward the surface of the sheet).

The information on migration gained from these experimental tests has been put to work by theoreticians Isaac Sanchez and Frank McCrackin in the form of mathematical migration models and decision rules. The purpose of these models is to provide a means for eliminating additive/oligomer combinations in polymers with high migration patterns without actually having to carry out many of the physical tests usually required.

"It was a very cloudy picture at the beginning," says Sanchez, "but gradually the key elements in the migration behavior of polyethylene began to emerge." It turns out, he says, that the two most important factors to be considered are the solubility of the additive or oligomer in the food simulating solvent and the diffusion rate of these molecules through the polymer.

For instance, if the solubility of the additive or oligomer is very low in the food-simulating solvent, it may be that even after an infinite length of time only a tiny amount will be absorbed by the food. And if the diffusion rate through the polymer is very slow, then it may be that by the time the product is sold and eaten only an insignificant amount will have reached the food.

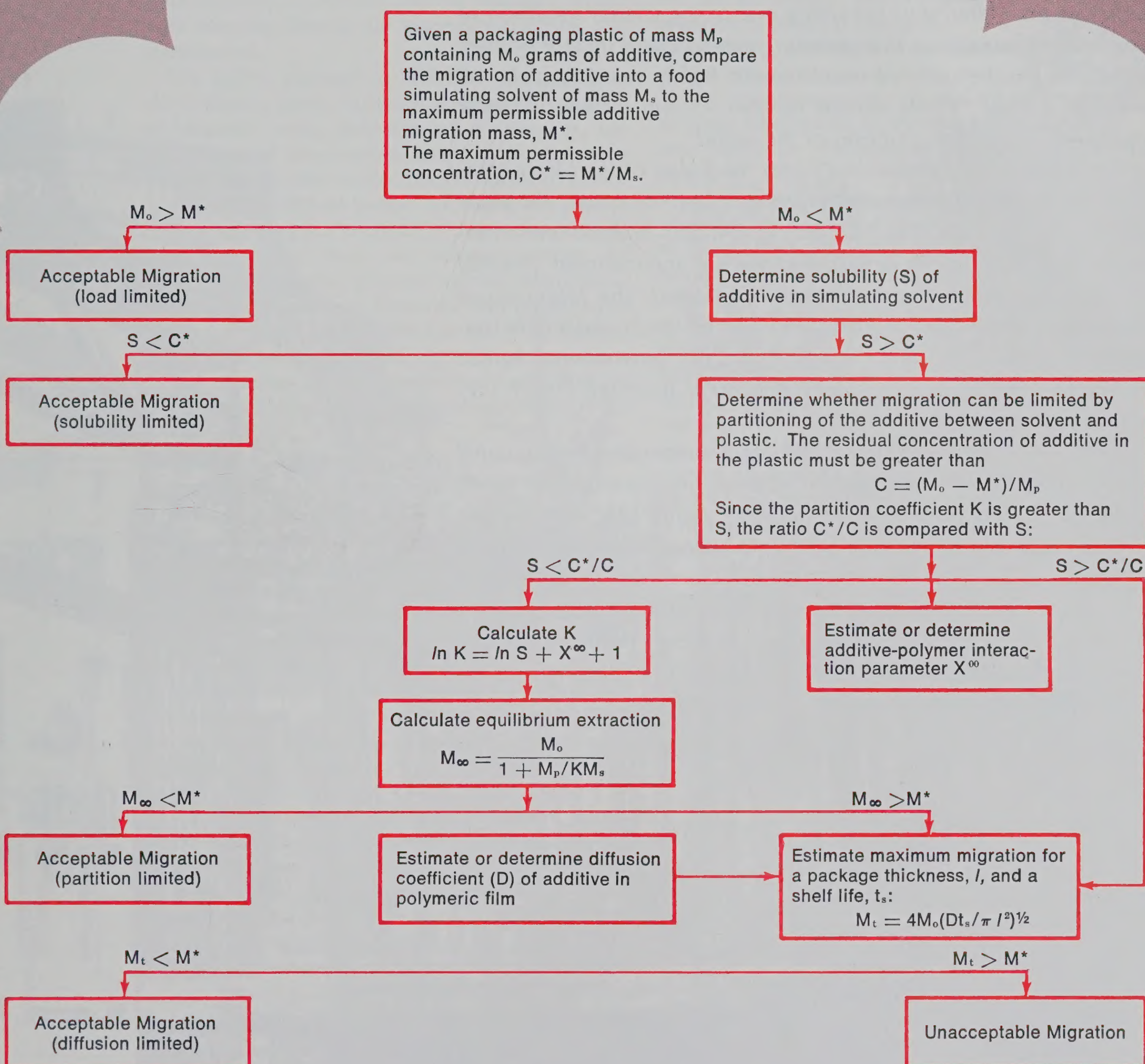
By applying thermodynamic theories to the migration problem, the Polymer Stability and Standards group has developed a decision tree (see diagram) for regulators and industry researchers that provides a step-by-step system for estimating "worst case" migration behavior. If the worst case migration estimates yield acceptably low migration levels, then there is no need for a full range of analytical tests.

This decision tree and the new migration equations the group has developed to go along with it may also prove helpful in predicting the migration of small molecules from synthetic materials used in orthopedic and other types of medical implants. These new tools can also be useful in determining the durability lifetime for polymers in general, based on the projected migration of protective additives.

In the future, the group will be conducting a similar range of tests and research to develop migration models for glassy polymers, such as polystyrene, commonly used in making egg cartons and hot beverage cups. Combining data from the polystyrene experiments with those on polyethylene, the researchers hope to produce a "generalized approach" to migration testing which both the FDA and industry would like to have.

In the meantime, says Smith, "there is no question of the safety and efficacy of the way FDA has regulated food packaging. Our purpose is just to provide the scientific framework that can make the process easier and more reasonable." Which rings a lot like the immortal words of a now forgotten scholar who said, "Science, after all, is the art of making things simpler." □

Migration Decision Tree



Report from the INTERNATIONAL BUREAU OF WEIGHTS & MEASURES

SUMMARY OF 1979 ACTIONS

Official standards of weights and measures, maintained for the United States since 1901 by the National Bureau of Standards, are linked to those maintained by the International Bureau of Weights and Measures in Sèvres, France. In fact, this international connection predates NBS. It was in 1875 that the U.S. Government signed the Treaty of the Meter, thus becoming one of the charter members of the treaty organizations that develop, maintain, and update primary standards for the units of measurement legally recognized by all member nations. Those nations number 45 and include all technologically advanced countries of the world.

The organizations established under the Treaty of the Meter comprise the General Conference on Weights and Measures, the body that makes final decisions on matters related both to the International System of Units (SI) and to the international physical standards by which those units are maintained; the International Committee for Weights and Measures, the technical and administrative arm of the General Conference; and the International Bureau of Weights and Measures (abbreviated BIPM from the French version of the name).

BIPM is the primary technical facility for cooperative international research and the meeting place for all treaty organizations. Dr. Pierre Giacomo, current director of BIPM, is the author of a recent report in Metrologia on the meeting held in October 1979 by the General Conference. Dr. Giacomo has graciously permitted DIMENSIONS/NBS to publish excerpts from his text. The following excerpts were selected and slightly edited by Louis Barbrow, former long-time member of the NBS technical staff and currently a consultant to the Office of Weights and Measures.

THE 16th General Conference on Weights and Measures (CGPM) took place in Paris, France, October 8-12, 1979. Delegations from 38 of the 45 nations that are signatories of the Treaty of the Meter attended. Dr. Ernest Ambler, Director of NBS, headed the U.S. delegation to this intergovernmental conference, and Dr. Edward L. Brady, NBS Associate Director for International Affairs, also participated.

The CGPM adopted six technical resolutions on the following items: kilogram standards, realization of electrical units, definition of the candela, improvement of photometric accuracy, special name sievert, and symbol for the liter. In addition, a resolution on the 1981-84 budget for CGPM was passed.

The following is a summary of the technical resolutions adapted from an account written by Dr. Pierre Giacomo, Director of the International Bureau of Weights and Measures, and published in the January 1980 issue of *Metrologia*. (The December 1978 DIMENSIONS/NBS reported on the actions of the meetings of the International Committee for Weights and Measures (CIPM) held September 19-22, 1978.)

Resolution 1. Kilogram standards

The 16th CGPM, *considering* that

• perfect conservation of kilogram standards (in particular, platinum iridium prototype kilograms) is the basis for accuracy of mass measurements,

• the transmission of this accuracy to kilogram standards made of other materials, such as stainless steels, is limited by an insufficient knowledge of the air buoyancy correction,

• this air buoyancy correction is usually calculated on the basis of ambient conditions, but that there are significant differences in formulas used among different laboratories,

recommends that

• laboratories study the effects of ambient conditions upon kilogram standards and the methods of improving conservation, and

• these laboratories intensify work aimed at an improved determination of the air buoyancy correction.

Resolution 2. Realization of electrical units

The 16th CGPM, *considering* that

• more accurate realizations of the SI units, particularly the volt and ampere, are important for all applications of electrical measurements,

• recent improvements have been made in these realizations directly and also indirectly by determination of physical constants,

• divergent results have been obtained,

• the origins of these divergences need to be resolved by comparison of independent realizations,

• improvement in accuracy will be attained when there is a better coherence among the different realizations of these units,

recommends that research be continued and intensified both on direct realization of electrical units and on the indirect realization through the determination of physical constants.

Resolution 3. Definition of the candela

The 16th CGPM, *considering* that

• excessive divergences still exist among the results of some laboratories in the realization of the candela based on the present "blackbody" primary standard,

• radiometric techniques are developing rapidly, allowing precisions not formerly possible,

• these techniques have enabled some national laboratories to realize the candela without construction of a blackbody,

• the value adopted by the CIPM in 1977 to relate quantities of photometry and radiometry (namely, 683 lumens per watt for the spectral luminous efficacy of monochromatic radiation of frequency 540×10^{12} hertz) has been accepted as sufficiently accurate for luminous photopic quantities, with a change of only about 3 percent required for luminous scotopic quantities, thus ensuring continuity,

recognizing that the time has come to define the candela in such a way as to allow improvement in the ease of realization and in the precision of photometric standards applied to both photopic and scotopic quantities, as well as those yet to be defined in the mesopic field,

defines the candela as the luminous intensity, in a given direction, of a source that emits monochromatic radiation of frequency 540×10^{12} hertz and of which the radiant intensity in that direction is 1/683 watt per steradian,

abrogates all previous definitions.

Resolution 4. Improvement of photometric accuracy

The 16th CGPM *instructs* the CIPM to organize international comparisons in order to check the uniformity of photometric measurements obtained by using the definition of the candela,

invites national laboratories to intensify their work aimed both at the realization of the candela following the new definition and at the improvement in photometric calibration procedures and methods of international comparisons.

Resolution 5. Special name sievert

The 16th CGPM, *considering* that

• there has been an appreciable effort to introduce SI units into the field of ionizing radiations,

• a risk to human beings of an underestimated

radiation dose could result from a confusion between absorbed dose and dose equivalent,

• the proliferation of special names represents a danger for the Systeme International d'Unites and must be avoided in every possible way, but that this rule can be broken when it is a matter of safeguarding human health,

adopts the special name sievert, symbol Sv, for the SI unit of dose equivalent in the field of radioprotection. The sievert is equal to one joule per kilogram.

Resolution 6. Symbol for the liter

The 16th CGPM, *recognizing* the general principles for writing symbols of units adopted in 1948 in Resolution 7 of the 9th CGPM,

considering that

• the symbol l for the unit liter was adopted by the CIPM in 1879 and confirmed in 1948,

• the name liter, although not included in the Systeme International d'Unites, must be admitted for general use with the System,

• in order to avoid the risk of confusion between the letter l and the number 1, a number of countries have adopted the symbol L for the unit liter,

decides to make an exception and adopt both symbols, l and L, for the unit liter, but

considering that only one of these could be retained in the future,

invites the CIPM to follow the development of the use of the two symbols and advise the 18th CGPM as to the possibility of suppressing one of them.

Meeting of the International Committee for Weights and Measures

Prior to the CGPM sessions, Ambler attended the 68th Session of the CIPM, held October 4-5, 1979, in Sèvres, France. The BIPM Director presented his annual report on activities of the BIPM since the 67th Session. He highlighted such topics as the preliminary results of an intercomparison of line scales, various problems posed by primary frequency standards used as clocks for the derivation of International Atomic Time, and current research on improving the maintenance of the volt and the ohm.

Proceedings of the 6th Session of the Consultative Committee for the Definition of the Meter (CCDM), held June 5-7, 1979, were reported by its President, D. Kind. Results of two intercomparisons were given. The first was a satisfactory limited intercomparison, among only four laboratories, of six end standards ranging in length from 0.45 m to 1.22 m. Results of the second intercomparison, albeit provisional, were much less satisfactory.

The following CCDM recommendation was subsequently approved by the CIPM:

Recommendation M 1 (1979). Line standards

The CCDM, *recognizing* the continued importance of line standards for length metrology,

recommends that research be pursued to improve the precision of line standards, in particular to improve the quality of both the lines and the surface upon which they are ruled, and that another intercomparison be undertaken after improved standards are available.

At CIPM's request, CCDM included angle measurement in its activities and initiated an intercomparison of angle standards. Eleven laboratories expressed an interest in participating and the National Research Laboratory of Metrology (Tokyo) agreed to act as organizer of the activity.

The CCDM also discussed the results of new measurements of laser wavelength and frequency (up to 380 THz) and found them to be compatible with those that previously led to the adopted value for the speed of light. Measurements of stability and wavelength had been made on argon ion lasers and He-Ne lasers, both stabilized on saturated absorption lines of iodine. The CCDM agreed that both of these laser radiations could be used as secondary standards of length with those already cited in Recommendation M 1 (1973).

An exchange of views among members of CCDM in the form of a BIPM questionnaire concerning an eventual change in the definition of the meter and subsequent discussion resulted in the CCDM concluding that the change, although inevitable, was not yet urgent. Some measurements of optical wavelength ratios are estimated already to be more accurate than the wavelength values expressed in meters. Inconsistencies could easily arise in the most precise length measurements, since the present system, which tends to be based upon the speed of light and four reference wavelengths, is greatly overdetermined.

To encourage all concerned laboratories to study the repercussions of a change in definition of the meter and to make the necessary preparations, the CCDM submitted the following recommendation to the CIPM:

Recommendation M 2 (1979). Definition of the meter

The CCDM, *considering* that

- the present definition of the meter does not provide a realization that is sufficiently precise for all needs and that therefore the definition should be changed,
- radiations exist which are more reproducible and more convenient than that specified by the present definition, that they are already in use as wavelength standards, but that their number continues to increase and it is not reasonable to expect that we can designate one as preferable,
- it has been agreed that it is desirable to preserve as the value of the speed of propagation in vacuum of electromagnetic waves, $c = 299\ 792\ 458$ m/s, recommended for use by Resolution 2 of the 15th CGPM (1975) in conformity with Recommendation M 2 adopted in 1973 by the CCDM,
- the relation between the wavelength in vacuum of a radiation, its frequency, and c leaves the possibility of choosing by convention only two of the following three elements: the definition of the meter, the definition of the second, and the value of c,
- the realizations of the present definition of the second are extremely precise, and that frequency is independent of geometrical parameters and therefore a quantity better suited to precise realization and measurement than is wavelength,
- in consequence, it is preferable to have the definition of the second and the value of c fixed by convention rather than to have, as at present, the definitions of the second and of the meter fixed by convention,
- experimental methods are already available for the measurement of the wavelength of a number of radiations useful for length measurement based upon the definition of the second and a better value of c, that these methods already furnish a precision better than that obtainable by using the standard radiation from krypton-86 and that methods still more precise are being studied, which take advantage of the progress made in lasers and the measurement of high frequencies,
- the CCDM will be able to propose the adoption of recommendations for the practical application of such a definition of the meter,

recommends that laboratories pursue research to improve optical frequency standards and the accuracy of their frequency in order to provide working standards which could be recommended by the CCDM for a realization of the following proposed new definition of the meter (to be proposed to the CGPM in 1983):

"The meter is the length equal to the distance traveled in a time interval of 1/299 792 458 of a second by plane electromagnetic waves in vacuum."

The CIPM subsequently approved this Recommendation in principle and sent it to the Consultative Committee on Units (CCU) for advice on the final form of an eventual redefinition of the meter.

The second consultative committee report at the CIPM session concerned the activities of the 8th session of the Consultative Committee for the Standards of Measurement of Ionizing Radiations (CCEMRI), July 9-10, 1979, with Ambler presiding. Section I of three CCEMRI sections reported on establishment of a working party to study the conversion factors required in deriving calibrations in terms of absorbed dose in water from calibrations in terms of exposure and from other data. It was recommended that the next two years of study provide refined numerical values for those conversion factors and that investigations be made of other possible methods for achieving absorbed dose-calibration in water. In addition to continuing comparisons of standards of exposure and absorbed dose, Section I decided to undertake a comparison of Fricke-type chemical dosimeters which are widely used at higher dose levels.

Section II reported very satisfactory intercomparisons of radioactivity measurements of ^{134}Cs sources. The 24 participating laboratories showed a standard deviation of 0.15 percent. Some other limited comparisons of ^{137}Cs were conducted with the results not quite so satisfactory. Investigations were extended to include new comparisons, such as of pure α- or β-emitters of electron-capture radionuclides.

Section III, Neutron Measurements, headed by Randall S. Caswell, Chief of the NBS Nuclear Radiation Division, received a report on comparisons of rapid monoenergetic neutron fluence rates now published in *Metrologia 16*, 31–49 (1980). A study of detectors as transfer instruments has resulted in preference for specific fission chambers in the use of direct counting instruments and in particular reactions, depending on energies, for methods based on activation. A recommendation proposing a 14-meV neutron dosimetry facility at BIPM was made, but CIPM postponed consideration on this pending a review of major expenditures and developments foreseen for the coming years. □

COVER STORY

The Sounds of Failure

DEVELOPMENTS IN ACOUSTIC EMISSION RESEARCH

by Michael Baum

KKKkkeeeeerrrrrroooooouuuuuaaaaacccccckkkkk. It always happens in those old Westerns, down in the abandoned gold mine, just after the hero has Found Something—that awful crack from overstressed roof beams just before the cave-in. Everyone in the audience recognizes the sound with an atavistic shudder.

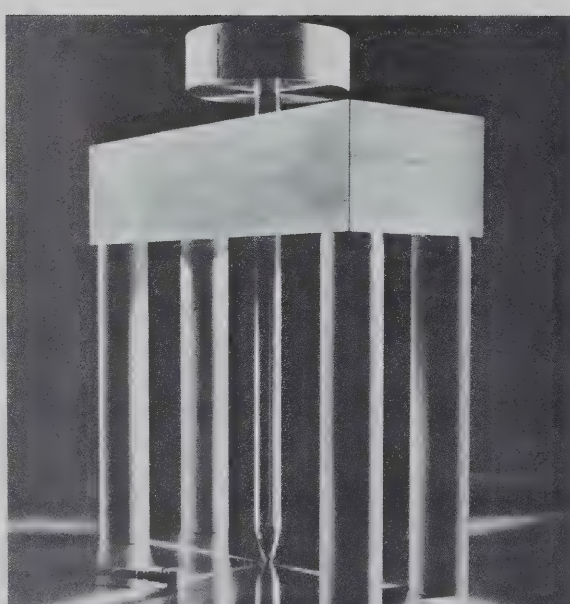
In the intervening years since the Gold Rush, the slight, ominous sounds that signal the impending failure of something important have acquired scientific respectability, technological sophistication, and a name—*acoustic emissions*, and acoustic emission (AE) research is one of the hot new areas in the growing field of nondestructive evaluation (NDE).

An acoustic emission is a sound wave—or perhaps more properly, a stress wave—that travels through a material as a result of some sudden release of energy in the material. A minute crack in a pressurized gas pipeline suddenly grows a bit longer, for example, and sends a stress wave through the pipe. In principle, if you were listening for it, you could tell from the sound where it originated in the pipe and what caused it.

In practice, it's not that simple, but Donald Eitzen, who coordinates an interdisciplinary research team on acoustic emission work at the National Bureau of Standards, argues that the potential benefits of the technology are great.

"One of the big advantages of the acoustic emission method," he says, "is that it is capable of continuously monitoring structures. You mount sensors on a structure and have them listen for an 'event' that occurs in the material. Most NDE techniques involve hauling in equipment, making measurements, and assuring yourself of the integrity of the structure at that particular point—but not in the indefinite future. Acoustic emission techniques—potentially, at any rate—allow you to monitor continuously things like nuclear reactor vessels."

"Potentially" is an important word, for there is still a lot of work to be done on a problem that is, as NBS engineer Nelson Hsu says, like "one black box inside another black box inside another black box."



Built to produce well-characterized acoustic emission signals, this apparatus consists of a diamond indenter (similar to those used to test the hardness of materials) that is pressed against the polished steel plate beneath it. The resulting sound waves are picked up by transducers similar to the one on the left.

Tin Cries and Missiles

Acoustic emission testing is a fairly young field, as such things are reckoned. It's true that people

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have always known an appropriate response to the creak of overloaded support beams, and that centuries of tinsmiths have known about "tin cry," the audible squeak made by tin under stress, but the first really quantitative study of an acoustic emission phenomenon did not appear until the late 1940's in a paper on the "tin cry" phenomenon by Mason, McSkimin, and Shockley of Bell Laboratories.

The Mason paper introduced the uses of piezoelectric sensors and electronic instruments to study the phenomenon, techniques which were used by the German, Joseph Kaiser, in the early 1950's to do the first comprehensive study of acoustic emissions in a variety of metals.

All of this stimulated a good deal of research on the ways acoustic emissions could be used to check critical structures for otherwise invisible flaws, and by 1964 the first paper appeared on the actual application of acoustic emission techniques to product testing. (This was in the testing of Polaris missile chambers.) The work grew out of research done for the Government by Aerojet General.

Since then acoustic emission techniques have been applied to petrochemical structures (pipelines, North Sea oil platforms, for example), airplanes (the Air Force uses acoustic emission sensors to monitor key points in the frame of KC-135 tanker planes), bridges, power plant components, and integrated circuit packages, to name a few.

Today the most successful uses of acoustic emission testing are in cases where one either has a good idea of where the failure points will be (and thus where to position the sensors) or what the most likely defects are. Typical examples are the pre-service testing of pressure containers and detecting leaks in pipelines.

Cataloging the applications of acoustic emission detection over the past 15 years gives the impression of a fairly well-developed technology, but in fact, much acoustic emission testing is still basically trial-and-error, as Hsu points out.

"The problem with acoustic emission is that when you detect something—some sound—then you know that something has happened, and you know *when* it happened, and if you are using several sensors you can use triangulation to know about *where* it happened. Knowing *what* happened is the art."

That is, of all the squeaks, snaps, and jolts that are likely to travel through a structure, how do you tell which are important and which are trivial? "Take the case of a nuclear reactor vessel," says Eitzen, "which is a fairly noisy environment. You want to be very careful to discriminate among signals. We can use acoustic emissions now in some very specific applications—monitoring strictly local conditions—but in general the technology is still being worked on."

It was considerations like this that led, four years ago, to the joint acoustic emission research project funded by NBS and the Electric Power Research Institute (EPRI). Results since then, ranging from theoretical advances in understanding the mechanism of acoustic emission effects to the development—just instituted this past January—of a new NBS calibration service for acoustic emission sensors, have brought the art of acoustic emission testing a lot closer to a science.

Black Boxes

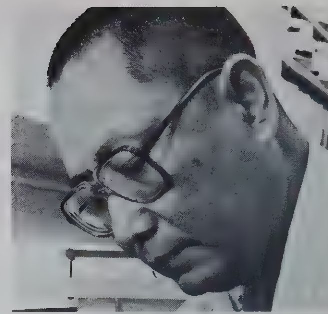
Why the National Bureau of Standards? Because most of the questions in acoustic emission testing today involve measurement problems. Picture yourself as an inspection engineer examining the data from an AE sensor attached to, say, a bridge girder. Typically what you see is a jagged graph of displacement versus time—a line with occasional peaks and plateaus which is supposed to represent how the metal under the sensor moved when a stress wave went through it. The shape of the stress wave is, in theory, determined by the nature of the event producing it. The sound of a crack growing in the girder is different from the sound of the metal bending under a load.

But how can you tell what the *original* sound was? One of the things you don't know is how much distortion is introduced by the sensor itself. Then, too, the sound wave picked up by the sensor has traveled through the girder and has been distorted by a variety of factors—internal reflections, the shape of the girder, changes in the density of the material, and so forth—into the signal that you see. More unknowns.

You can check the performance of the system by introducing a stress wave of your own—dropping a ball bearing on the girder, for example—but, unless you know the characteristics of *that* sound and how it will be modified fairly well, all you've done is to add another unknown.

This is the situation Nelson Hsu was referring to when he described acoustic emission work as a nested set of "black boxes."

"There are two approaches to this problem," says Hsu, "the empirical and the analytic. Most of the industry has used the empirical approach. Generate sounds, listen to what you get, then vary some-



NBS acoustic emissions engineer Dr. Nelson Hsu.

thing and listen again. Western Electric, for example, has been very successful with this."

However, Hsu explains, the empirical technique is not always practical. To return to an earlier illustration, the cost would be prohibitively high to build nuclear reactor pressure vessels just to study the acoustic emissions of various flaws in various locations under various conditions.

So NBS turned to a more analytic approach—trying to find some answers to general questions about the basic mechanisms of acoustic emission. "The first thing NBS did," says Hsu, "was to break down the problem—separate the boxes."

One of the first efforts, in the mid-1970's, was to record undistorted AE signals from simulated "events." Oddly enough, this was something which had not really been done before. The basic problem was that the sound waves traveling through a piece of metal reflected from the edges or faces of the structure. Eventually the structure was set into resonance like a plucked string. In addition, the transducer introduced resonances of its own. As a result, the recorded sounds tended to emphasize the natural or resonant frequencies of the structure rather than the original sounds. "We looked at the standard acoustic emission collection techniques and found a lot of faults," says physicist Franklin Breckenridge, who, along with Carl Tschiegg and Martin Greenspan, pioneered this work at NBS. "The typical sound spectra obtained by these techniques showed many resonances, and these were much more related to the structure and the transducer than to any property of the emission event."

Of course, if the piece of metal were infinite in size, there would be no reflections from the edges, and if the transducer didn't touch the surface, there'd be no reflections from the transducer.

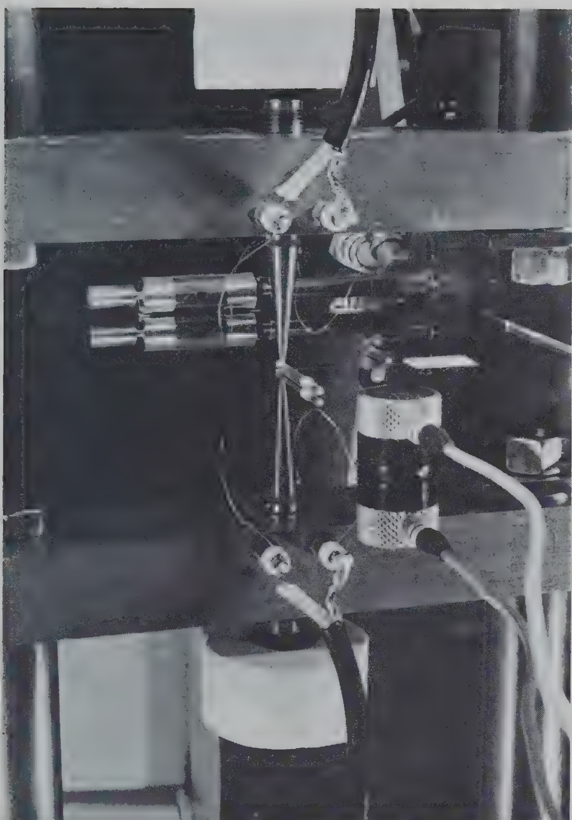
Breckenridge and his coworkers decided on a test block in the shape of a thick plate with the signal source mounted on one face and the transducer on the opposite face. The sound wave from the source arrived at the transducer first and the reflections arrived later, too late to contaminate the data. This arrangement acted, in effect, like an infinite block of metal. They also devised a special transducer—based on a variable capacitance technique—that did not resonate or affect the block appreciably because it worked with only the slightest contact with the metal.

Given a simple test structure and a new type of transducer, a third requirement was to find a source of sounds similar to acoustic emissions that could be reproduced at will. Originally the idea was to find a source with a comparatively simple waveform to check the performance of the transfer block and capacitive-transducer arrangement. The answer—which has since become widely used in the industry—was the breaking of a glass capillary.

The researchers checked several sources of stress waves and decided that the best thing to do would be to put a short piece of glass capillary on top of the metal plate and slowly tighten a screw down on it. There would be an initial load, with a sudden release in pressure when the capillary snapped. The AE signal would look like a "step function"—a sudden sharp rise followed by a level line.

As it turned out, the received waveform didn't look quite like the physicists expected, "because we didn't expect the right thing," Breckenridge notes reasonably. The source produced a nice step function, as expected, but even the simplest metal structure modified the waveform. As it turned out, when the proper theory was applied to the transfer block, the received signal was found to be quite accurate, and the breaking glass capillary did provide a clean, easily reproduced source of AE signals.

The techniques learned in this experiment proved to be valuable. The capillary, capacitive transducer, and "transfer block" have since become the components of the new NBS system for the calibration of AE transducers. The original plate, an aluminum disc about 6 cm thick and 18 cm in diameter, has



Left. Close up of the glass capillary AE source shows a typical piezoelectric transducer (foreground), a length of a thin-walled glass capillary under the indenter (center), and the capacitive transducer used as a reference (background). The wires on the indenter are attached to a load cell which measures the force released when the capillary breaks.

Right. Franklin Breckenridge adjusts indenter pressure on the glass capillary device he developed. The massive steel block beneath the apparatus was ground and polished to a nearly optical quality flat by the NBS Optical Shop, "an impressive feat in itself," says Breckenridge.

since been scaled up to a polished cylinder of steel, 90 cm in diameter and 4 cm thick—a gift from Bethlehem Steel.

Meanwhile, Bureau specialists in the study of materials were looking at acoustic emission questions from their own point of view. Researchers under physicist Edwin Fuller began a project in parallel with the acoustics group to create stress events in a transfer block and study the signals. The twist was that while the acoustics experts were trying to create an idealized source signal, the materials experts tried the experiment with an idealized transfer block—a thick glass plate.

"Glass is an ideal elastic material," explains Fuller, "It's isotropic—that is, a block of glass has the same physical properties in any direction—and homogeneous. There is little attenuation, and scattering of the stress waves is less of a factor than in a metal plate. If you can't get it to work in glass, it would be hopeless to try it in something else, like a metal alloy, and in any event, the ideal case should be the proof of the theory."

The materials specialists also developed their own ideas for stress "events," including, in one case, a

sort of laboratory air gun that fired small particles at the glass plate.

Listening Backwards

While the development of these new experimental systems was an important step, part of the NBS contribution to acoustic emission research was more intangible—the development of theory.

One of the central theoretical problems in acoustic emission research is to determine a mathematical function, known as the "Green's function," which defines how the original source signal is changed by the characteristics of the structure it passes through.

Given the Green's function analysis of a particular arrangement of signal source, structure, and transducer, you can take a specific source signal and calculate what the signal detected by the transducer will be. More importantly, given the signal detected by the transducer, if you can find the inverse function, you can use it to calculate what the original signal was, and this is important, for in all the years of AE research, very few *source* signals actually have been determined. "If you know the Green's function for a geometry," says Eitzen, "you have the whole solution in your hands—the solution to any problem in that class."

For this reason, it was an important accomplishment when three men, Professor John Willis of the University of Bath (then visiting the United States), NBS mathematician John Simmons, and Hsu worked out first the theory and then the computational procedures for the Green's function for an infinite metal plate.*

There would still be no infinite-sized metal plates, but as we have seen, Breckenridge's disc would act like one for the crucial few microseconds.

In 1976, for the first time, a theory was used to *calculate* the result of sending a signal from a particular source (the breaking glass capillary) through a particular structure (the aluminum transfer block). The subsequent experiment agreed almost perfectly with the theory.

In 1977, also for the first time, the theory was used in the opposite direction to calculate the input function from the signal picked up by the transducer. The source used was a breaking lead in a mechanical pencil (see box). "Now no one," Hsu remarks, "is interested in infinite plates, and no one is interested in breaking pencil leads, and capacitive transducers are still difficult to use, but since we now have a complete, well-known, closed-loop system, we can substitute new factors one at a time and determine the results."

A good example of this use of the experimental AE system is the recently established NBS calibration service for acoustic emission transducers. The typical AE transducer used in industry relies on the piezoelectric effect: a pressure sensitive crystal is attached to the structure to be monitored, and the stress waves from the AE source generate an electric current in the crystal which can be detected and measured. Although piezoelectric transducers can be even more sensitive than the capacitive transducers used in the NBS set-up, their behavior—how they respond at different frequencies and amplitudes—is considerably less well-known. So the new calibration service—the first of its kind—is an important step in improving the accuracy and reliability of acoustic emission testing in the field.**

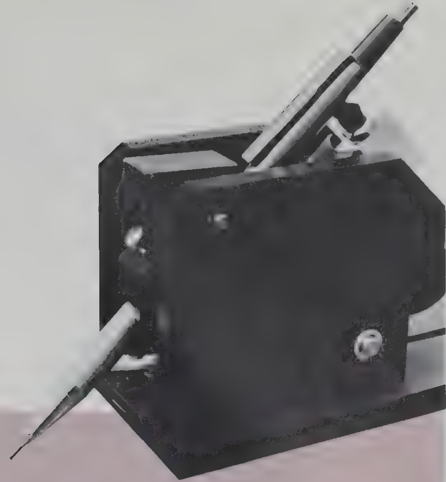
Much of Hsu's time of late has been spent applying the AE system to a much more complex theoretical problem called *time domain deconvolution*—a mathematical technique he used to analyze an acoustic emission signal recorded at some distance from the source and to reconstruct the characteristics of the original signal. In principle at least, and to some extent now in fact, Hsu's work will make it possible to tell from the AE signal, as picked up by some transducer, the nature of the original event.

Part of the inspiration for this work, says Hsu, was a recording he heard in 1978, when he started the project, of the long-dead operatic tenor Enrico Caruso. The only recordings of Caruso's voice ever made were done with fairly primitive equipment which—like most AE systems—distorted the original sound. An enterprising company had applied similar deconvolution techniques in an attempt to recapture the sound of Caruso's voice, with, according to Hsu, fairly respectable results.

Sophisticated deconvolution techniques are also central to work being done now by metallurgist Roger Clough and mathematician John Simmons. Clough and Simmons are pioneering the use of multiple transducers and deconvolution mathematics to "image" acoustic emission sources,—once again with an eye toward distinguishing one

**The theoretical solution was found independently, it should be noted, by Professor Y. H. Pao and his students at Cornell University. See: Pao, Y. H.; Gajewski, R. R.; and Ceranoglu, A. N.* Acoustic Emission and Transient Waves in an Elastic Plate. *J. of Acous. Soc. of A. v. 64. (1978)*

***For information concerning this service, contact: Franklin R. Breckenridge, B147 Sound Building, National Bureau of Standards, Washington DC 20234. Tel. 301/921-3646.*



Above. Designed by Nelson Hsu, this simple AE source is built around a common mechanical pencil. The stand holds the pencil at a fixed angle and includes a load cell which measures the pressure at which the pencil lead breaks.

Serendipity

Most pieces of precision testing equipment cost hundreds or thousands of dollars. One instrument used by Nelson Hsu, however, costs only a couple of bucks. It's a mechanical pencil.

Hsu happened on the idea in 1975 while a consultant to Lockheed Georgia. He was looking for a variant of Breckenridge's glass capillary apparatus, a device to furnish a clean, reproducible, *useful* signal source for calibrating acoustic emission equipment or making experiments. A mechanical pencil, it occurred to him, was just the thing for holding a glass capillary—run the capillary out of the pencil to the desired length, press it against the appropriate piece of metal, and snap. Besides, every engineer owned one.

The idea caught on fast—was, in fact, patented by Lockheed—so one little side investigation Hsu started was a systematic study of various things to put into the mechanical pencil. The goal was something readily available in thin rods that could always be counted on to break the same way, with approximately the same force, and to produce the same acoustic signal in a given piece of metal. After about a year of testing everything from various types of glass capillaries to boron fibers, Hsu and his co-workers decided on the best material. Right—the pencil leads that come with the pencil.

Hsu has since developed the idea into a true precision instrument, fitting the pencil in a stand that holds it at a precise angle and guarantees that the pressure on the point is applied in a smooth, even manner. It is also instrumented with pressure sensors so the researcher can measure the magnitude of the signal directly. Of course all that (plus inflation) has jacked up the price.

Left. Metallurgist Roger Clough with diamond indenter apparatus.

Right. Physicist Dr. Edwin Fuller with test set-up used to study acoustic emission phenomena in glass plates. The tube under the horizontal glass plate is actually a compressed gas air gun which fires small particles at the plate. The top surface of the glass is covered with gold plate to provide an electrically conductive surface, which is instrumented with a capacitive transducer.

event from another. The use of several transducers at the same time is not new; it's a common way to locate AE sources by triangulation, explains Clough, "but that's not really using all the information that's available. When you go to the deconvolution problem—actually studying the nature of the source—that's another order of magnitude in difficulty."

Clough's descriptions are peppered with terms from the geophysics vocabulary, like "epicenter" and "Rayleigh wave", and indeed, he says, the relationships between NBS work and the study of earthquakes are quite close, "The developments in both fields are closely parallel."

The Wave of the Future

Current AE research at the Bureau of Standards runs in a variety of directions, depending on the interests of the researcher. Part of the effort is now going into refining the measurement system. On the boards are improved transducers—both capacitive and piezoelectric—for measuring horizontal and vertical vibrations, studies of the effects of the shape of a transducer on its performance, development of experimental techniques to determine the Green's function for more complicated structures, and work on the algorithms and techniques for doing deconvolution of more complicated systems.

Another line of research uses acoustic emission techniques to study properties of materials. Fuller and his colleagues have found that the techniques developed to study AE events in glass are particularly useful in studying erosion processes in ceramics—work with applications to problems as varied as the damage to airplane components in dust storms and the breakdown of turbine blades in coal gasification systems.

"You can look at erosion as an impact event," says Fuller, "multiple impacts at high velocity in a short time frame. These are particles measured in micrometers and there is no way to measure the force when one hits the surface, but since it *is* an impact, it sends stress waves through the body, and from *those* you can get an idea of the original event and calculate the force. As it turns out, you can take a static particle and just push it into the surface and get cracks and fractures similar to those from a moving particle. Using acoustic emission techniques we can make measurements down to microsecond loading times."

Another project in the same group is an attempt to use acoustic emission techniques to test ceramic chip capacitors for the Navy. These particular capacitors, which go into missile guidance systems, represent something of a mystery. Every so often a capacitor which looks normal and passes standard electrical tests will fail after it is installed on a circuit board and put into use. The current best guess is that there are microscopic flaws in some capacitors that only become apparent under certain stress conditions. The NBS researchers are attaching AE transducers to the capacitors and breaking pencil leads against them to cause them to "ring." They have discovered that each capacitor has an individual AE spectrum—a sort of signature—and they hope to discover some sort of correlation between capacitors that fail and certain types of signatures.

Clough and his co-workers are using AE as a particularly sensitive way to study the fracture and phase transitions of different materials. They have coupled AE detectors to a small diamond indenter—a device commonly used to measure the hardness of a material by the amount of force needed to push a small pyramidal diamond into the surface. A sufficiently brittle material will develop microcracks under the diamond which create acoustic emissions that can be detected. The idea, now being patented, is to build a small, hand-held field device which can be used by inspectors to monitor the degradation of materials in hostile environments like coal conversion plants.

Acoustic emission is admittedly still somewhat in its infancy, but the researchers in the field are united in believing it has great potential. A good capsule summary is given by Haydn Wadley, scientist and coworker of Simmons and Clough, visiting NBS from the British Atomic Energy Research Establishment, "Acoustic emission should enable you to do two things. First, not only to locate cracks but to learn other things, like the size and orientation of the crack, the area it covers, and the speed and direction in which it is growing.

"Second, it can be applied to the study of fracture and deformation processes, which over the past 20 years has been limited by the lack of a dynamic technique—especially one that works at speeds close to the speed of sound. We may ultimately be able to apply acoustic emission to the study of phase transformation processes in metals, such as solidification studies."

Who knows? When future adventure tales are written, maybe, *before* going into the uranium mine, the hero will pause to stick a portable AE transducer on the roof beam.

NONDESTRUCTIVE EVALUATION PROGRAM ANNOUNCED

In an effort to improve nondestructive testing methods used in aerospace manufacturing and to strengthen ties between private industry and government research, the National Bureau of Standards and the Martin Marietta Corporation have begun two 6-month cooperative programs.

Both programs provide for the exchange of personnel. Harold Berger, chief of the NBS Office of Nondestructive Evaluation located in Gaithersburg, Md., has been visiting several Martin Marietta Aerospace manufacturing plants, reviewing in depth the corporation's methods of nondestructive testing and exchanging information about possible ways to upgrade industry testing and quality control capabilities. James Beal, a group engineer for Martin Marietta Aerospace at New Orleans, La., is now working with researchers in the NBS Electrical Measurements and Standards Division on the development of an eddy current inspection system capable of imaging surface flaws and, to a limited degree, subsurface flaws in metals.

Nondestructive evaluation (NDE) techniques make use of high frequency sound waves, electrical currents, x-rays, or other phenomena to check the integrity of materials and manufactured products without destroying or damaging the items being examined. In the manufacture of missiles, aircraft, nuclear reactors, and other high technology products, nondestructive testing is a critical element in assuring the quality of production.

"Practically everything industry uses and welds requires nondestructive testing," says John Saunders, director of manufacturing for Martin Marietta Aerospace. This can be a time-consuming and expensive aspect of manufacturing.

Consequently, the availability of appropriate standards to improve the efficiency and accuracy of nondestructive tests may greatly affect manufacturing costs. "I hope," says Berger, "to get a good appreciation for what a major company like Martin Marietta is doing in NDE, so that we at NBS can direct our industrial standards work in the most useful manner."

Although NBS managers have kept abreast of industry NDE usage through brief visits and a variety of joint meetings, the 6-month duration of this program should provide a more realistic view of a major manufacturer's day-to-day operations, according to Berger.

In turn, Saunders expects that Berger's suggestions based on NBS research experience should help his company tailor its inspection systems to actual needs and identify promising research areas for the firm to investigate. "We are being given a lot of good advice," he says, "and I think there is a lot to be gained."

Beal's assignment at NBS, on the other hand, is part of the Bureau's Research Associate Program. The program enables scientists and engineers from industrial, professional, trade, and other organizations to work, under the sponsorship of their employers, at NBS research laboratories on projects of mutual interest to NBS and the sponsor.

One of Martin Marietta's current projects in which nondestructive testing is extremely important is a contract with the National Aeronautics and Space Administration (NASA) to build the external fuel tank for the Space Shuttle Program. The entire fuel tank, which measures 47 meters long and 8.5 meters wide, must be inspected carefully with nondestructive techniques. The tank is now under construction at NASA's Michoud manufacturing facility in New Orleans.

Beal is Martin Marietta's principal researcher for the development of rapid production inspection systems for the space shuttle tank's 912 meters of aluminum welds. While working at NBS, Beal hopes to learn more about eddy current flaw detection systems, which may provide a less expensive, more convenient way of inspecting welds than the radiographic and ultrasonic techniques used presently.

Eddy current inspection techniques, which rely on changes in a material's electrical conductivity to detect flaws, are already used widely in industry. However, NBS researchers are now developing a new eddy current imaging system capable of displaying real-time pictures of flaws. An imaging system of this type would greatly reduce the time required to interpret eddy current data, while improving the reliability of the technique.

In the NBS device, electrical currents are fed through coils set at right angles to the metal being inspected. This creates a magnetic field which in turn sets up circular (eddy) currents within the metal. Any flaws or cracks at or near the surface of the metal will cause variations in the magnetic field generated by the eddy currents. These variations are detected at the metal's surface by separate sensing coils.

Beal is now working with the NBS researchers to determine characteristics of subsurface flaws by using this device and imaging the detected flaws directly on a television screen.

While at the Bureau, he also will be conducting a literature search of methods that may prove useful in the development of a "deep penetration" eddy current imaging system. The development of an eddy current system able to resolve clearly subsurface flaws in metals would eliminate the chief disadvantage associated with the NDE technique.

In addition to this program with Martin Marietta, there are 29 other research associate programs, involving 96 individuals, currently in progress at the Bureau. More than 400 organizations and 1000 individuals have participated in the Research Associate Program since it was established in 1921. G.P.

STAFF REPORTS

BOND ENERGIES AND CHEMICAL REACTIVITY

Bond energies are the essential ingredients for understanding chemical reactivity. Recent studies at NBS suggest that many long accepted values are in error. It is demonstrated that the new bond energies provide a sound basis for understanding cis-trans isomerization and small ring decyclization processes.

Wing Tsang, Chemical Kinetics Division, A145 Chemistry Building, 301/921-2792.

The course of any chemical reaction is marked by breaking and forming chemical bonds. For a quantitative understanding of chemical kinetics, a knowledge of the appropriate bond energies is necessary. Chemical kineticists usually define the bond dissociation energy as the enthalpy of reaction for the process $AB \rightarrow A + B$ at 298 K, where A and B are molecular fragments. Note that this is a thermodynamic quantity and is directly related to the enthalpies of the molecule and the fragments.

However, since A and B are frequently unstable, direct determinations through heat of combustion measurements are not feasible. Indirect techniques must be used for all except the smallest fragments, which means kinetic methods. Thus, one selects a reaction involving the unstable species in question and measures the rate of the reaction in both directions. Detailed balancing yields an equilibrium constant which can then be used to determine the heats of reaction if structural information for all species is known. Alternatively, a heat of reaction can be calculated from the temperature dependence of the equilibrium constant as measured across a range of temperatures. For all these measurements the instability of the molecular fragments introduces severe uncertainties and the process of determining heats of formation or bond energies is necessarily fraught with assumptions. For bond energies to play a role in predictive theories, accuracy requirements are extremely stringent.

Compound	New NBS values (kJ/mol)	Previous values (kJ/mol)
Ethyl-H	420	412
Isopropyl-H	407	395
N-Propyl-H	420	409
S-Butyl-H	407	398
I-Butyl-H	422	417
T-Butyl-H	404	387
T-Amyl-H	405	
Cyclohexyl-H	416	395
Allyl-H	377	369
Isobutenyl-H	373	
1-Methylallyl-H	375	355
Propargyl-H	384	393
3-Methylpropargyl-H	385	
Benzyl-H	372	352

For example, in the cases where the bond energy is directly reflected in the rates for a particular process, an error of 6 kJs will result in an order of magnitude deviation in rate at 298 K and a factor of 2 at 1000 K.

The history of gas kinetics has been marked by repeated determinations of bond energies and, as new techniques and understandings have developed, by continual revisions of what had been thought to be well established numbers. Recent work at NBS has led to new insights on such measurements. The validity of this work is suggested by the self-consistency of the results as derived from a wide variety of measurements. Specifically, the NBS studies have concentrated on the reactions:

$$A_2 \underset{k_b}{\overset{k_f}{\rightleftarrows}} 2A$$

(where A = ethyl, isopropyl, and t-butyl radicals). These studies have shown that high-temperature determinations carried out at NBS for k_f at 1100 K and a reinterpretation of low-temperature measurements are completely consistent with existing results on the reverse reaction k_b. The combined studies cover a range of rate constants of 35 orders of magnitude. Thus, for these "over-determined" systems we arrive at a consistent set of thermal properties for the unstable species A. Furthermore, the calculated entropies agree with present day ideas on the molecular structure of the species involved. The enthalpies are, however, significantly different from those determined by scattered measurements of the type described earlier.

The NBS results are thought to be more reliable due to the larger data base and the consistency of determinations. Generalizing from these results, we arrive at the values for the heat of formation of simple organic radicals presented in table 1. Also included for comparative purposes are previously recommended values. It should be noted that the general trend of the new data is toward increasing bond energies. In other words, most aliphatic hydrocarbons are much more stable than previously assumed.

Table 1—*Comparison between old and new values for carbon-hydrogen dissociation energies.*

An interesting consequence of these findings, which serves to illustrate the use of bond energies as a predictive tool, deals with the isomerization of small ring hydrocarbons. This is a subject of great current interest for which a considerable volume of data exists. The study of such highly strained systems pushes chemical theory to its limits. The simplest way of visualizing such processes is to postulate a biradical intermediate formed from the breaking of the highly strained C-C bond, followed by appropriate rotations and finally recyclization. The activation energy for the isomerization is therefore: $E = \Delta H_T$(ring opening) $+ RT$, and ΔH_T(ring opening) $= 2D_T$ (C-H) $- D_T$ (H-H) $+ \Delta H_T$ (hydrogenation), where D_T is the bond dissociation energy at temperature T and the heat of hydrogenation refers to the conversion of the cyclane to the appropriate aliphatic compound. Since the last two terms are accurately known, the dependence of the activation energy on the bond dissociation energy is quite clear. Such calculations performed by using the older bond dissociation energies have always resulted in values approximately 20- to 40-kJ lower than experimentally measured. As a result, it has been necessary to postulate a 20- to 40-kJ activation barrier for recyclization or rotation. Structural and quantum mechanical considerations are, however, at variance with the assumption of such a barrier, making this subject quite controversial. With the new bond energies, we obtain the activation energy results summarized in table 2. It can be seen that the deficit has been almost completely removed and in most cases is probably within the experimental uncertainty. Thus, it is now clear that the biradical mechanism does provide an accurate quantitative basis for predicting the activation energy for the isomerization of small ring compounds.

Compound	Experimental (kJ/mol)	From NBS bond energies (kJ/mol)	Previous results (kJ/mol)
Ethylene (2-membered ring)	275	272	255
Cyclopropane	268	255	238
1,2 Dimethylcyclopropane	259	263	250
1,1,2,2, Tetramethylcyclopropane	228	232	188
1,2 Dimethylcyclobutane	257	251	230
Vinylcyclopropane	208	208	180

Table 2—*Comparison between predicted and experimental activation energies for ring opening reactions.*

NUCLEAR FUEL ASSAY USING RESONANCE NEUTRONS

Researchers in the Center for Radiation Research are developing a technique that will produce images showing the distribution of any isotope desired. These images will be similar to the familiar x-ray image. Using neutrons generated by the NBS linear accelerator, this technique—resonance neutron radiography—provides a unique tool for determining both distribution and content of any sample. The technique is being developed as a reference standard measurement system for the NBS Office of Measurements for Nuclear Technology, which seeks to provide accurate and timely methods for keeping track of nuclear materials. The resonance neutron radiographic technique can be used to determine the concentration and uniformity of distribution of fresh fuel rods and to measure nuclear material in scrap and waste from a nuclear plant.

Roald A. Schrack, James W. Behrens, and Charles D. Bowman, Nuclear Radiation Division, B119 Radiation Physics Building, 301/921-2677.

Resonance neutron radiography represents a new application and use for information that has already been accumulated. The physical phenomenon that is utilized in the technique is the selective absorption and scattering of neutrons by the nucleus. Every isotope has a different set of energies for which it interacts with neutrons. These energies have been carefully cataloged so that the observations of a characteristic absorption pattern can be used to identify the presence of an element—and in particular the amount and isotope of the element. Previous research at NBS has determined to better than one percent accuracy the values of these interaction cross-sections for a wide variety of materials. These data were submitted to the national clearing house maintained at the Brookhaven National Laboratory on Long Island. We drew on these files in the analysis of our data.

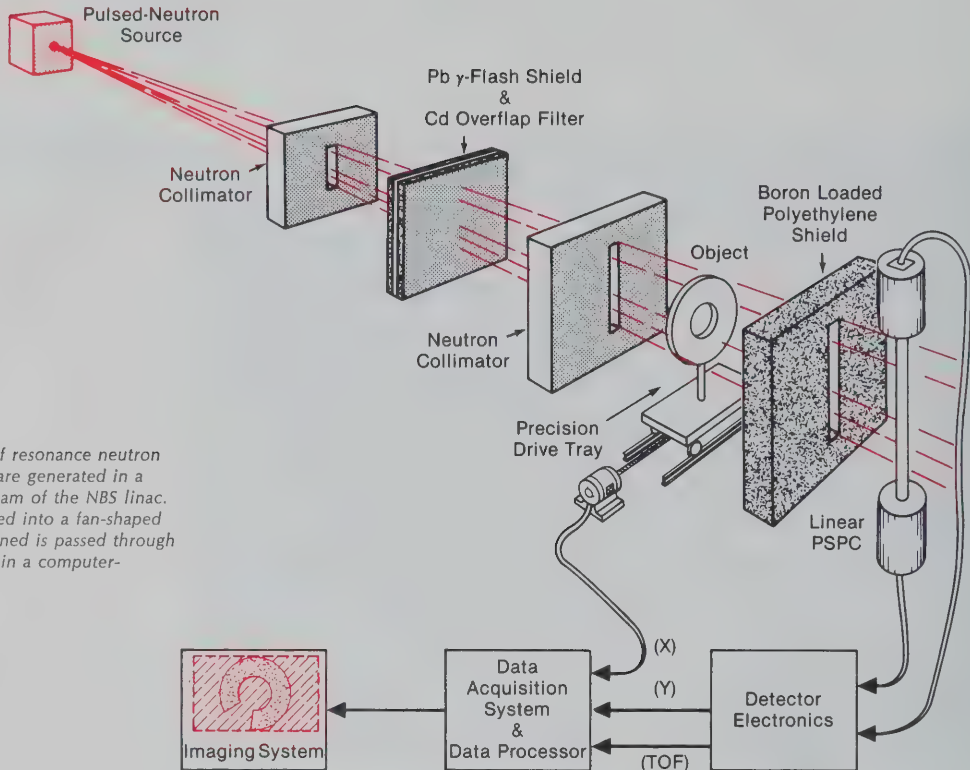


Figure 1—*Schematic diagram of resonance neutron radiography system. Neutrons are generated in a source by using the electron beam of the NBS linac. The neutrons are then collimated into a fan-shaped beam. The sample to be examined is passed through the beam to produce an image in a computer-controlled system.*

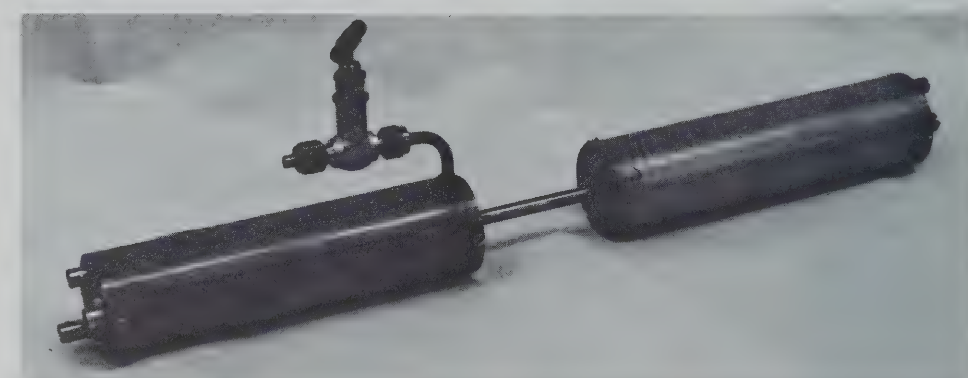


Figure 2—*High resolution, position-sensitive neutron detector. The detector region is the center small-diameter tube. Special amplifiers are enclosed in the large-diameter cylinders at each end. The valve allows the gas used in the ionization chamber to be changed to determine the effects of different mixtures and pressures. The detector is presently operating with a mixture of 3.1 x 10^5 pascals of ^3He, 7.9 x 10^5 pascals of xenon, and 0.4 pascal of carbon dioxide. (10^5 pascals ≃ 1 atmosphere).*

The procedure used is shown in figure 1. Neutrons generated by the NBS linear accelerator are collimated down to a thin fan-shaped beam. A sample of material to be analyzed is placed in this beam. The number and location of neutrons that pass through the sample are determined with a position-sensitive neutron detector placed behind the sample. This information is accumulated in a computer as the sample is scanned. When the sample is completely scanned, the accumulated data are analyzed to produce a picture of the distribution of the desired isotope in the sample. The total amount of the isotope in the sample can then also be determined.

The resolution of the detector system determines the researcher's ability to discern details in the distribution of the desired isotope. Commercially available detectors have a resolution limit of about 5mm; details smaller than this can not be observed. Fuel pellets used in nuclear reactors are 10mm in diameter, so the available resolution in commercial detectors does not permit a meaningful measurement of nuclear fuel. To overcome this deficiency, a high-resolution detector system was developed by NBS in coordination with the Oak Ridge National Laboratory. This detector, shown in figure 2, has a resolution of about 1 mm and has been used in the analysis of nuclear fuel.

In addition to its value in the Nuclear Safeguards Program, resonance neutron radiography promises to be of value in other fields such as nondestructive evaluation. The examination of manufactured items for hidden defects is frequently done with low-energy neutrons as well as x rays. Hidden defects in welds can be detected before the equipment is used, thus preventing costly equipment failure. As an example of this type of application, a sample was made with an intentionally defective joint. Figure 3 shows the result of a resonance neutron radiography of this sample when the distribution of silver

Figure 3—*Computer-generated distribution of silver in a defective silver solder joint as determined by a resonance neutron radiographic scan.*

in the brazing material was made. No defect was observed with normal x-ray imaging techniques. The defect was only marginally detectable using low-energy neutron imaging techniques.

Research is continuing on the resonance neutron radiographic technique to improve accuracy and resolution and to reduce the time required to produce an image. Although the technique is now restricted to laboratories with high-energy linear accelerators, methods are being worked on to allow the technique to be used on a broader basis and thus become more available as a tool for technology.

STANDARD FOR LEAD ON FILTER MEDIA

The Office of Standard Reference Materials at the National Bureau of Standards has recently issued a new Standard Reference Material (SRM) 2674, Lead on Filter Media, developed particularly for use in environmental analysis.

This new SRM is one of a series resulting from a program, jointly sponsored by the Environmental Protection Agency and the National Bureau of Standards, which is aimed at developing reference materials for use in monitoring the environmental atmosphere. The SRM is intended for use in the calibration of apparatus and the evaluation of methods used in the determination of atmospheric lead that has been collected on filters.

SRM 2674 consists of a series of glass fiber filter strips (16 mm x 203 mm) upon which lead nitrate has been deposited in an essentially central location. The values certified correspond to the quantities of the substance leached from the filter strip matrix. The nominal composition of the filter strips in the set are:

Sample No.	*Lead μg*
I	100
II	300
III	1500
Blank Filter	0

This SRM consists of two glass fiber filters at each of the levels indicated above. The filter material is typical of filter material used in high volume air samplers.

SRM 2674, Lead on Filter Media, may be purchased for $80 from the Office of Standard Reference Materials, B311 Chemistry Building, National Bureau of Standards, Washington, DC 20234; telephone 301/921-2045.

OYSTER TISSUE STANDARD REFERENCE MATERIAL

A marine animal tissue Standard Reference Material (SRM) is now available from the National Bureau of Standards. Certified for chemical composition, Oyster Tissue, SRM 1566, is intended for use in validating analytical determinations, calibrating the instrumentation used for such determinations, and referring the measurements to a common base.

In recent years, concern about contamination of marine animals used as foods has increased. To determine the extent of contamination, investigators need accurate data on the trace element composition of marine animals from various locations in clean and polluted aquatic environments. A number of marine animals, especially bivalves, are known to concentrate heavy metals and other substances from their aquatic environment. Some species of oysters and mussels exhibit this property to such a marked degree that scientists have proposed their use as sentinel organisms for monitoring pollutant levels in coastal marine waters. Accurate data on the pollutant levels in such bivalves can be obtained efficiently by analyzing the samples relative to a similar marine animal tissue in which concentrations have been certified for the elements of interest. Use of a common reference base, such as SRM 1566, by investigators enables intercomparison of results from various studies.

In the preparation of SRM 1566, freeze-dried oyster material was cryogenically ground, tested for homogeneity, and analyzed.

Concentration values are certified for 19 elements: arsenic, cadmium, calcium, chromium, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, rubidium, selenium, silver, sodium, strontium, uranium, and zinc. The concentrations of the following elements are given for information only: bromine, chlorine, cobalt, fluorine, iodine, molybdenum, phosphorus, sulfur, thallium, thorium, and vanadium. A number of analytical techniques were used for these analyses including atomic absorption spectroscopy, atomic emission spectroscopy, isotope dilution mass spectrometry, neutron activation, photon activation, and polarography.

This SRM was requested and partially funded by the Food and Drug Administration. Analyses for SRM 1566 were performed in the Inorganic Analytical Research Division; laboratories in Japan and Canada provided cooperative analyses.

Standard Reference Material 1566, Oyster Tissue, may be purchased from the Office of Standard Reference Materials, B311 Chemistry Building, National Bureau of Standards, Washington, DC 20234, for $65 per 30-gram sample.

CONFERENCES

For general information on NBS conferences, contact JoAnn Lorden, NBS Public Information Division, Washington, DC 20234, 301/921-2721.

HIGH RESOLUTION INFRARED APPLICATIONS AND DEVELOPMENTS

The design, calibration, and application of tunable infrared lasers was the focus of a conference held at the National Bureau of Standards in Gaithersburg, Md., June 23–24, 1980. Sponsored by the NBS Molecular Spectroscopy Division, the conference was partially supported by the Exxon Research and Engineering Company, the NBS Office of Standard Reference Data, and the NBS National Measurement Laboratory.

The purpose of this meeting was to bring together active workers from four areas:

New instrumentation design and fabrication

Wavelength and frequency calibration

High resolution molecular spectroscopic techniques, and

Applications (industrial, analytical, astrophysical, etc.)

While most discussions centered on the current state-of-the-art and promising future developments in tunable infrared laser activities, the conference also touched on these same aspects for activities utilizing Fourier transform instruments.

Well-recognized experts from General Motors Research Laboratories, the University of Waterloo in Canada, NASA-Goddard Space Flight Center, Kitt Peak National Observatory, University of Denver, Lincoln Laboratories, the Molecular Institute of Japan, and NBS presented formal papers in areas ranging from the spectroscopy of important chemical processes to measurements of planetary atmospheres by space probes.

A panel discussion was held each day of the conference on the topics of "Tunable Infrared Laser Sources," "Absolute Wavelength Measurement in the Infrared," and "Calibration Standards." Poster sessions, allowing a free exchange of information between conference participants on individual research findings, covered the related topics of "Tunable Source Instrumentation and Applications," "Fourier Transform Instrumentation and Application," "Molecular Spectroscopy," and "Calibration Techniques."

Researchers interested in further technical information about the conference should write to the conference chairman, Jon Hougen, Molecular Spectroscopy Division, NBS, Washington, DC 20234.

THIRD SEMINAR ON LINEWIDTH MEASUREMENTS FOR IC INDUSTRY

The National Bureau of Standards is planning the third in a series of special limited-attendance seminars on the accurate and precise optical measurement of 0.5- to 10-μm linewidths on integrated circuit (IC) silicon wafers and photomasks.

The 4-day meeting, to be held at the NBS site in Gaithersburg, Md., from July 15 to 18, 1980, will be open to a maximum of 30 engineers and senior technicians from the IC industry—IC manufacturers, photomask suppliers, and instrument manufacturers.

Present IC geometries require measurements in the 0.5- to 10-μm range. NBS-IC industry interlaboratory studies have found systematic errors in linewidth measurements as large as 1 μm between systems. Until the recent development of NBS Standard Reference Material (SRM) 474, a linewidth standard for optical measurements, there were no well-defined physical standards in this size range to ensure the accurate calibration of optical microscope systems.

The seminar on Linewidth Measurements on Integrated Circuit Photomasks and Wafers will be composed of lectures and laboratory sessions. The lectures will discuss the theory and problems associated with optical measurements of linewidths on photomasks in transmitted light and of linewidths in silicon dioxide on wafers in reflected light. The material dependence of calibrations will be stressed with emphasis on the differences that occur between measurements on thick and thin oxide-coated wafers. The laboratory sessions will provide "hands on" experience in performing calibrations with the NBS linewidth standard, SRM 474, in transmitted light and measurements of linewidths in thin oxides of silicon wafers in bright field reflected light.

Other topics that will be covered are: theory of the optical microscope, proper microscope operating conditions for accurate measurements, edge detection, data analysis, and transfer of accurate measurements from NBS to industry.

For further information contact Elaine Cohen, A305 Technology Building, NBS, 301/921-3786.

6TH INTERNATIONAL SYMPOSIUM ON NOISE IN PHYSICAL SYSTEMS

Noise in Physical Systems is the subject of a 5-day conference to be held at the National Bureau of Standards, Gaithersburg, Md., April 6–10, 1981.

The study of noise in a physical system provides insights into its microscopic

dynamic behavior. In addition to being a source of information, noise can also be a source of irritation in that it limits performance of devices. The study of noise is of prime importance for the testing of physical theories as well as for the development of improved physical measurements and improved performance of devices. The conference considered those theoretical and experimental studies which lead to better understanding of noise mechanisms in physical systems.

Electronic noise continues to be an important concern of the conference. In addition, contributions discussing noise in a wide variety of other physical systems were encouraged. A partial list of topics includes:

- **Mechanisms**

g-r noise, hot electrons, density fluctuations, phase fluctuations, multiplication noise

- **Specific systems**

FETS, Josephson junctions, plasmas, electro-optics, astronomical, size effects, magnets

- **l/f noise**
- **Quantum effects**
- **Applications and Measurement Techniques**

quality control, light scattering, thermometry

- **Theory**

nonlinear effects, localization, stochastic processes

For further information contact Kathy Stang, B348 Materials Building, NBS, 301/921-3295.

CONFERENCE CALENDAR

***June 16-21**

INTERNATIONAL ORGANIZATION OF LEGAL METROLOGY (OIML), DEPARTMENT OF STATE; sponsored by NBS, Dept. of State, and International Bureau of Legal Metrology; contact: David Edgerly, A353 Physics Building, 301/921-3307.

June 19

19TH ANNUAL TECHNICAL SYMPOSIUM ON PATHWAYS TO SYSTEM INTEGRITY, NBS, Gaithersburg, MD; sponsored by NBS and ACM; contact: Carol Wilson, A252 Technology Building, 301/921-3861.

June 22-27

NATIONAL CONFERENCE ON WEIGHTS AND MEASURES, Shoreham-Americana, Washington, D.C.; sponsored by NBS and NCWM; contact: Harold Wollin, A211 Metrology Building, 301/921-3677.

June 23-25

HIGH-RESOLUTION INFRARED APPLICATIONS AND DEVELOPMENTS, NBS, Gaithersburg, MD; sponsored by NBS; contact: Jon Hougen, B268 Physics Building, 301/921-2021.

July 22-24

NBS EMI METROLOGY SEMINAR, NBS, Gaithersburg, MD; sponsored by NBS; contact: M. Gerald Arthur, EMI/Radiation Hazards Group, Electromagnetic Fields Division, NBS, Boulder, CO; 303/499-1000, ext. 3603.

August 14-15

ASME SYMPOSIUM ON CRITICAL MATERIALS AND FABRICATION ISSUES, St. Francis Hotel, San Francisco, CA; sponsored by NBS and ASME; contact: Jeffery Fong, A302 Administration Building, 301/921-2631.

***September 22-24**

NATIONAL CONFERENCE OF STANDARDS LABORATORIES, NBS, Gaithersburg, MD; sponsored by NBS and NCSL; contact: Brian Belanger, B362 Physics Building, 301/921-2805.

September 29-October 1

CERAMICS AS ARCHAEOLOGICAL MATERIAL, NBS, Gaithersburg, MD; sponsored by NBS and Smithsonian Institution; contact: Alan Franklin, A355 Materials Building, 301/921-2901.

October 7-9

COAL CONVERSION, NBS, Gaithersburg, MD; sponsored by NBS and DOE; contact: Samuel Schneider, B308 Materials Building, 301/921-2894.

***December 10**

COMPUTER NETWORKING SYMPOSIUM, NBS, Gaithersburg, MD; sponsored by NBS and IEEE; contact: Ira Cotton, B226 Technology Building, 301/921-3516.

1981

***April 6-10**

6TH INTERNATIONAL SYMPOSIUM ON NOISE IN PHYSICAL SYSTEMS, NBS, Gaithersburg, MD; sponsored by NBS and the Catholic University of America; contact: Robert J. Soulen, B128 Physics Building, 301/921-2018.

***June 8-12**

SECOND INTERNATIONAL CONFERENCE ON PRECISION MEASUREMENTS AND FUNDAMENTAL CONSTANTS, NBS, Gaithersburg, MD; contact: Barry N. Taylor, B258 Metrology Building, 301/921-2701.

**New Listings*

PUBLICATIONS

WEIGHTS AND MEASURES GUIDE

*Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices, Nat. Bur. Stand. (U.S.), Handb. 44, 1979 Ed., 213 pages (Dec. 1979) Stock No. 003–003–02143–1, $6.**

The National Bureau of Standards, in cooperation with the National Conference on Weights and Measures (NCWM), has published a revised edition of NBS Handbook 44, *Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices.* This revised text includes all amendments to the 1971 edition of the Handbook through 1979.

Designed as a working tool for weights and measures officials, equipment manufacturers, installers, and repairmen, the Handbook describes the technical requirements recommended by the NCWM for use in commercial weighing operations. These requirements are intended to eliminate the use of inaccurate or faulty standards, weights and measures, and weighing and measuring devices.

Handbook 44 is the standard reference of the NCWM and, as such, is referenced in the weights and measures laws of many local jurisdictions. Local jurisdictions are encouraged to accept and use National Conference codes in order to provide a system of uniform requirements throughout the country.

**Publications cited here may be purchased at the listed price from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402 (foreign: add 25%). Microfiche copies are available from the National Technical Information Service, Springfield, VA 22161. For complete periodic listings of all scientific papers and articles produced by NBS staff, write: Editor, Publications Newsletter, Administration Building, National Bureau of Standards, Washington, DC 20234.*

BETTER DATA ENTRY METHODS, COST SAVINGS

Guidelines for Selection of Data Entry Equipment, Nat. Bur. Stand. (U.S.), Fed. Info. Process. Stand. Publ. (FIPS PUB) 67, 23 pages (1979).

Commercially available data entry devices and recommended procedures for comparing their cost and performance are described in a new publication issued by the National Bureau of Standards' Institute for Computer Sciences and Technology.

Published as Federal Information Processing Standards Publication (FIPS PUB) 67, the *Guideline for the Selection of Data Entry Equipment* is intended to aid Federal automatic data processing managers in selecting data entry equipment and in cutting the cost of data entry.

Data entry is the process of preparing data in a machine-readable form and entering it into a computer. While there have been recent improvements in techniques and equipment for data entry, these advances have not been as marked as advances in the speed of data processing. Moreover, since data entry is more labor-intensive than most information processing operations and often represents from 30 to 50 percent of an agency's total information processing costs, proper equipment selection can produce substantial savings in operating costs.

The guide analyzes features of many different data entry devices to help Federal managers select cost-effective methods from the large number of available systems. The operational requirements, costs, typical applications, and advantages and disadvantages of each kind of data entry equipment are reviewed.

Four basic equipment types are covered: (1) keying equipment such as keypunch and other keyboard devices; (2) automatic readers such as optical character reader and magnetic ink character recognition systems; (3) source data sensing devices such as voice recognition systems; and (4) miscellaneous devices such as point-of-sale terminals.

The guide recommends that Federal managers consider the use of advanced data entry methods that reduce labor and processing costs by producing tape, disk, or direct input to computers.

In comparing keying equipment, for example, the guide points out that newer electronic devices such as keyboard-to-tape and keyboard-to-disk systems can increase the volume of data entry about 20 to 50 percent over keypunch devices. This improved performance is due to software that automates some of the keying, checks the accuracy of entered data, and performs other tasks that simplify data entry.

Despite these special software features, keyboard devices that depend on operator entry for most of the data will be limited to a very modest data input rate, no matter how sophisticated the equipment. Therefore, long-term improvements in data entry will come from the use of automated data acquisition and entry techniques, according to the guide. These include devices such as point-of-sale terminals, process control instrumentation, and environmental measuring equipment, which collect data directly in machine-readable form without operator processing.

Detailed procedures are provided in the guide for analyzing data entry requirements and comparing equipment features. It recommends that factors such as cost/performance, system flexibility, user acceptance, and possible future system improvements be considered in selecting data entry equipment.

Background information on the subject can be found in *Selection of Data Entry Equipment,* Spec. Publ. 500–55, which may be ordered for $3.50. Order by Stock No. 003–003–02133–4 from the Superintendent of Documents, Government Printing Office, Washington, DC 20402.

NBS FIRE RESEARCH OUTLINED IN NEW PUBLICATION

Third Annual Conference on Fire Research (NBSIR 79-1916) may be ordered for $12 from the National Technical Information Service, Springfield, VA 22161. Order by #PB 80–110240.

From the physics and chemistry of fires to the behavior of people in fire emergencies, the fire research activities of the National Bureau of Standards are summarized in a new publication now available.

Programs of the Bureau's Center for Fire Research and outside research carried out under NBS contracts and grants are covered in the 186-page report, entitled *Third Annual Conference on Fire Research*. Also described are activities of the extramural Fire Research Program, NBS-funded research projects carried out by 30 universities and research groups. This research supports and supplements internal programs of the Center, which is the Federal Government's primary fire research laboratory.

The report provides basic information about investigations of smoldering combustion, smoke and radiation, ignition and inhibition, product flammability, fire detection and control systems, enclosure modeling and fire spread, human behavior patterns in fires, and a range of other fire-related topics. The details provided are based upon the presentations at the August 22–24, 1979, conference. The publication includes abstracts of all of the NBS Center's internal and extramural activities, thus providing an overview of the entire program.

PUBLICATIONS LISTING

Analytical Chemistry

Dunstan, L. P., Gramlich, J. W., Barnes, I. L., and Purdy, W. C., Absolute Isotopic Abundance and the Atomic Weight of a Reference Sample of Thallium, J. Res. Nat. Bur. Stand. (U.S.), *85* No. 1, 1–110, (Jan.-Feb. 1980).

Building Technology

Olmert, M., and Raufaste, N., Eds., Building Technology Project Summaries 1979, Nat. Bur. Stand. (U.S.), Spec. Publ. 446–3, 82 pages (Feb. 1980) Stock No. 003–003–02150–4, $4.

Computer Science and Technology

Branstad, M. A., Cherniavsky, J. C., and Adrion, W. R., Computer Science and Technology: Validation, Verification, and Testing for the Individual Programmer, Nat. Bur. Stand. (U.S.), Spec. Publ. 500–56, 26 pages (Feb. 1980) Stock No. 003-003-02159–8, $1.75.

Consumer Information and Protection

Wollin, H. F., Ed., Model State Laws and Regulations, Nat. Bur. Stand. (U.S.), Handb. 130, 1979 Edition, 107 pages (Feb. 1980) Stock No. 003–003–02152–1, $4.50.

Electromagnetic Metrology

Kenney, J. M., Semiconductor Measurement Technology: Modulation Measurements for Microwave Mixers, Nat. Bur. Stand. (U.S.), Spec. Publ. 400–16, 86 pages (Feb. 1980) Stock No. 003–003-02154–7, $3.75.

Electronic Technology

Jerke, J. M., Ed., Semiconductor Measurement Technology: Accurate Linewidth Measurements on Integrated-Circuit Photomasks, Nat. Bur. Stand. (U.S.), Spec. Publ. 400–43, 166 pages (Feb. 1980) Stock No. 003–003–02151–2, $5.

Energy Conservation and Production

Gass, S. I., Ed., Validation and Assessment Issues of Energy Models. Proceedings of a Workshop held at the National Bureau of Standards, Gaithersburg, Md., Jan. 10–11, 1979, Nat. Bur. Stand. (U.S.), Spec. Publ. 569, 559 pages (Feb. 1980) Stock No. 003–003–02155–5, $9.50.

Measurement Science and Technology Physical Standards and Fundamental Constants

Howe, S. L., Ed., NBS Time and Frequency Dissemination Services, Nat. Bur. Stand. (U.S.), Spec. Publ. 432, 20 pages (Sept. 1979) Stock No. 003–003–02105–9, $1.50.

Schoonover, R. M., Davis, R. S., Driver, R. G., and Bower, V. E., A Practical Test of the Air Density Equation in Standards Laboratories at Differing Altitude, J. Res. Nat. Bur. Stand. (U.S.), *85*, No. 1, 27–38 (Jan.–Feb. 1980).

Nuclear Physics and Radiation Technology

Loftus, T. P., Standardization of Iridium-192 Gamma-Ray Sources in Terms of Exposure, J. Res. Nat. Bur. Stand. (U.S.), *85*, No. 1, 19–25 (Jan.–Feb. 1980).

Standard Reference Materials

Velapoldi, R. A., and Mieleng, K. D., A Fluorescence Standard Reference Material: Quinine Sulfate Dihydrate, Nat. Bur. Stand. (U.S.), Spec. Publ. 260-64, 139 pages (Jan. 1980) Stock No. 003–003–02148–2, $4.25.

Thermodynamics and Chemical Kinetics

Wu, Y. C., and Young, T. F., Enthalpies of Dilution of Aqueous Electrolytes: Sulfuric Acid, Hydrochloric Acid, and Lithium Chloride, J. Res. Nat. Bur. Stand. (U.S.), *85*, No. 1, 11–17 (Jan.–Feb. 1980).

NEWS BRIEFS

NBS LAB STUDIES VERIFY HEALTH HAZARDS TO FIRE FIGHTERS. NBS is working with the U.S. Fire Administration to inform the fire fighting community about some research results and potential hazards associated with certain fire conditions. Tests at the Bureau's Center for Fire Research show that phosgene gas is generated when polyvinyl chloride (PVC), a material commonly used in electrical wiring insulation, is subjected to electrical arcing. Other NBS studies have confirmed that burning of urea formaldehyde foam, used recently as thermal insulation in walls of new and existing homes, produces substantial quantities of deadly hydrogen cyanide gas. This adds further weight to the argument that fire fighters should be especially careful to use breathing apparatus during and for some time after a fire is put out.

NEW TECHNIQUE MEASURES CORROSION UNDER COATINGS. Researchers at NBS have developed a new technique for studying the corrosion of metal under organic coatings. The new method combines two corrosion measurement strategies--ellipsometry and pH changes--into a single, very sensitive apparatus for nondestructive monitoring of coated metal in corrosive environments. Preliminary studies at the NBS Center for Materials Science indicate that the method has application as a basic research tool, both for unraveling the complicated chemistry needed to explain the failure of paints and other organic coatings and for systematic laboratory testing of various anti-corrosion paint systems containing chemical inhibitors.

NBS DEVELOPS AN "INVISIBLE SENTINEL." A method for detecting swimmers and waders in shallow water environments, such as rivers, bays, and marshes, has been developed by NBS for the Department of Defense. Employing an acoustic interferometric technique, the method could be used to detect the presence of intruders around docks, bridges, and shore installations. A major advantage of the system is its freedom from precision positioning of the source and sensors, allowing rapid installation even by untrained personnel. An additional advantage is its relatively low cost.

FIRST GRAPHICS EXCHANGE SPECIFICATION. NBS has recently released a specification facilitating the exchange of graphic or geometric information from one computer-aided design/computer-aided manufacturing (CAD/CAM) system to another. Developed in cooperation with several Federal agencies (Army, Navy, Air Force, and NASA), this specification, called the Initial Graphics Exchange Specification (IGES), also allows for archiving of data found in different systems. A method for data exchange was seen as a major necessity for realizing the full potential of computer technology advances. A company or organization can now develop codes to translate data between any two systems through the intermediate step of IGES.

INVENTORS' WORKSHOP SERIES ANNOUNCEMENT. Workshops are being held in five U.S. cities to help inventors develop and market their inventions. Special emphasis will be on energy-related ideas, but the program will be directed at all inventors. These workshops are sponsored by DOE, NBS, and the American Association of Engineering Societies, which represents nearly one million engineers throughout the country. The Series was initiated in San Francisco, May 17-18; it continues in Boston, June 20-21; Atlanta in September; Dallas-Fort Worth, October 31-November 1; and Philadelphia in November. Atlanta and Philadelphia dates will be announced when they are set.

NEXT MONTH IN

DIMENSIONS NBS

You are a jetliner pilot coming in for a blind landing at a fog-shrouded airport. The bottom light on your beacon indicator flashes purple . . . on your headset you hear tones like the busy tone on a telephone receiver . . . you cannot see the end of the runway . . . Are you on course for a blind landing, or should you apply power, pull up, and try again? July DIMENSIONS/NBS gives some insight into use of instrument landing systems (ILS).

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Luther H. Hodges, Jr., Deputy Secretary

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NATIONAL BUREAU OF STANDARDS
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The Commerce Department's National Bureau of Standards was established by Congress in 1901 to advance the Nation's science and techonology and to promote their application for public benefit. NBS research projects and technical services are carried out by the National Measurement Laboratory, the National Engineering Laboratory, and the Institute for Computer Sciences and Technology. Manufacturing, commerce, science, government, and education are principal beneficiaries of NBS work in the fields of scientific research, test method developments, and standards writing. DIMENSIONS/NBS describes the work of NBS and related issues and activities in areas of national concern such as energy conservation, fire safety, computer applications, materials utilization, and consumer product safety and performance. The views expressed by authors do not necessarily reflect policy of the National Bureau of Standards or the Department of Commerce.

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Annual subscription: Domestic, $11.00, foreign, $13.75. Single copy: Domestic, $1.10, foreign, $1.40. The Secretary of Commerce has determined that the publication of this periodical is necessary in the transaction of the public business required by law of this Department. Use of funds for printing this periodical has been approved by the Director of the Office of Management and Budget though June 30, 1981.

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